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Title: A TECHNIQUE FOR MEASURING COMPRESSIVE PROPERTIES OF SINGLE MICROBALLOONS: COMPARISON OF CARBON MICROBALLOONS OF VARYING TAP DENSITIES

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A Technique for Measuring Compressive Properties of Single Microballoons*

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A technique has been developed to obtain mechanical properties of individual hollow microspheres, or microballoons. This technique utilizes a nanoindentation instrument equipped with a cylindrical sapphire tip, thereby replicating a conventional mechanical compression test on a nanometer scale. The procedure has thus been termed nanocompression, since the extreme sensitivity of the nanoindentation instrument provided a load resolution of 50 nN and a displacement resolution better than 0.02 nm. The load-displacement curves resulting from this test provided mechanical properties including maximum load and strain to failure. Materials tested included polymer, glass, and carbon microspheres, with the primary focus being carbon microballoons. Characterization of the microballoons, in terms of wall thickness and diameter, was undertaken through quantitative microscopy in an effort to correlate morphology to mechanical properties. A trend has been observed between strain to failure and carbon microballoon diameter.

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A Technique for Measuring Compressive Properties of Single Microballoons: *Comparison of carbon microballoons of varying tap density*

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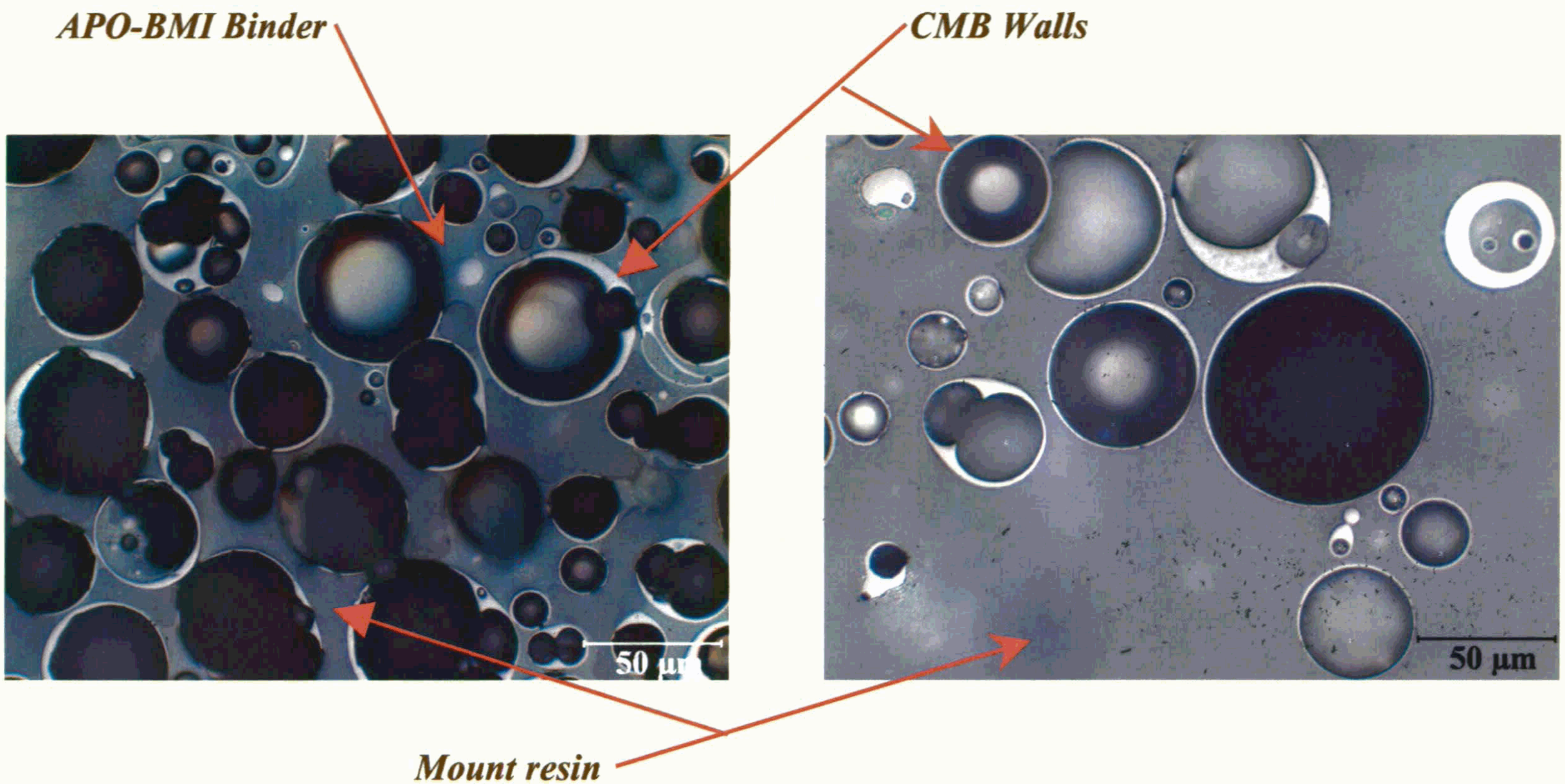
Carbon microballoons: Materials, morphology, and significance

Materials

- Carbon microballoons (CMB) produced from phenolic microballoon precursors via carbonization
- CMB of three tap densities:
  - 0.143 g/cm^3
  - 0.161 g/cm^3
  - 0.177 g/cm^3
- Tap density
  - Per ASTM B 527 - 93
    - Measure volume occupied by known mass of MB after 3000 taps
    - ρ_{tap} = mass/settled volume

CMB morphology

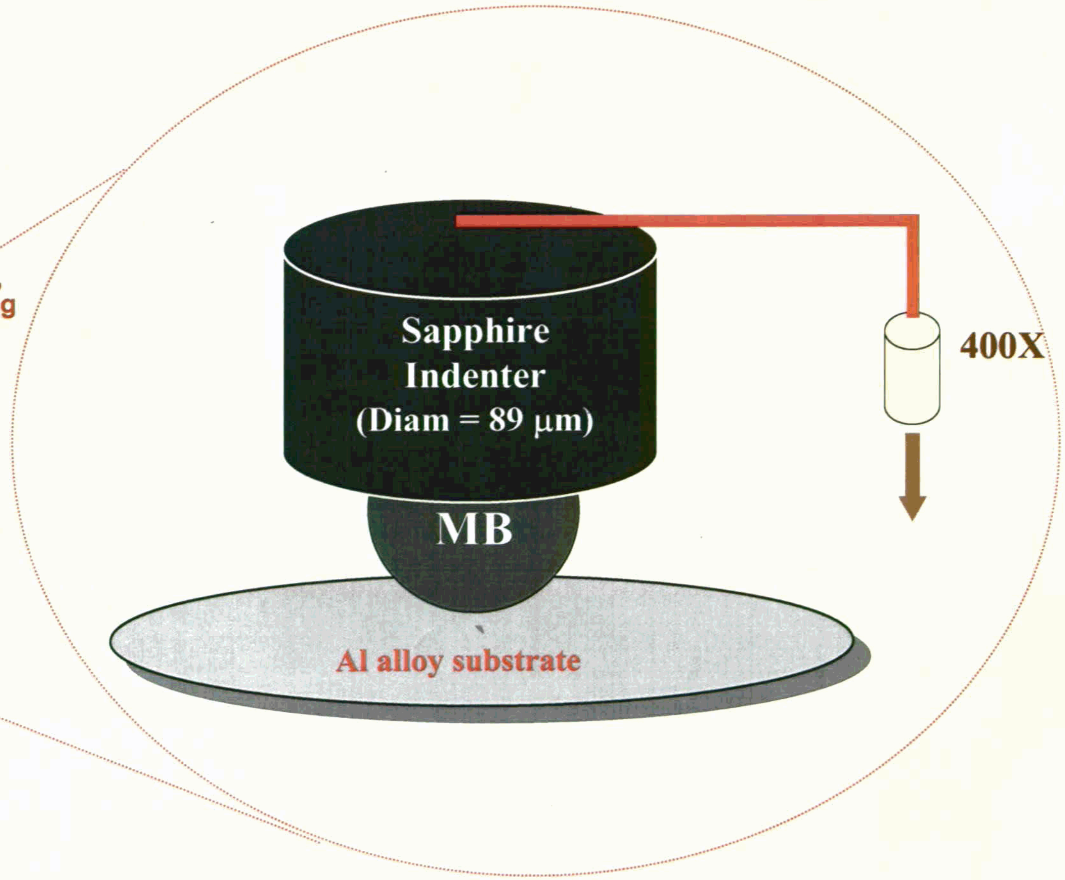


- Single walled spheroidal CMBs or CMBs with multiple compartments (Nested CMBs)

Experimental procedures

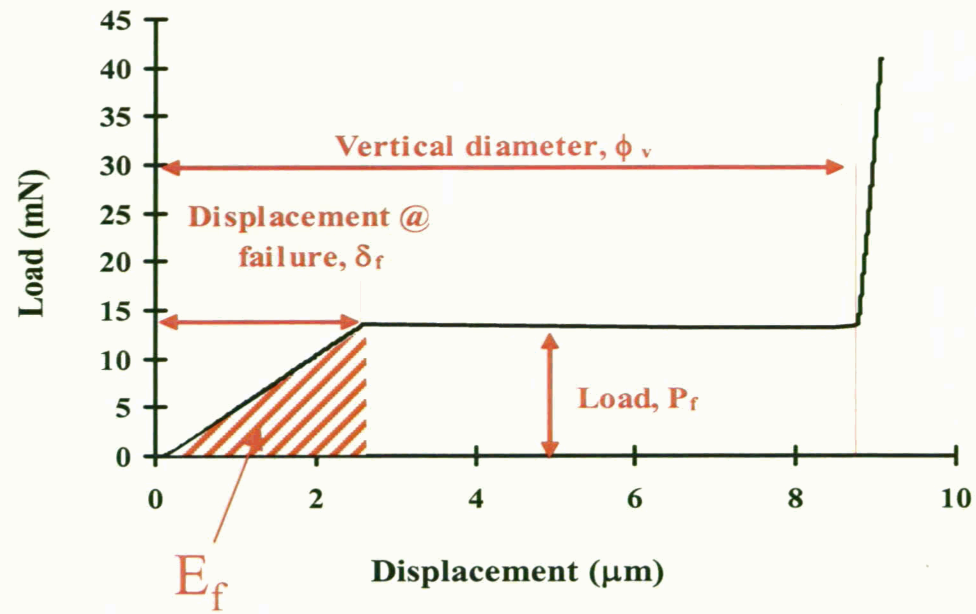
Procedure: Nanocompression test

- Nanoindenter XP II system specifications
  - Displacement resolution
    - < 0.02 nm
  - Maximum indentation depth
    - 500 μm
  - Maximum load
    - 500 mN (50.8 g)
  - Load resolution
    - 50 nN (5.1 μg)
  - Maximum displacement
    - 2 mm
  - Position control
    - Remote with joystick (motorized in x-, y-, and z- directions). Fully automated during experiments

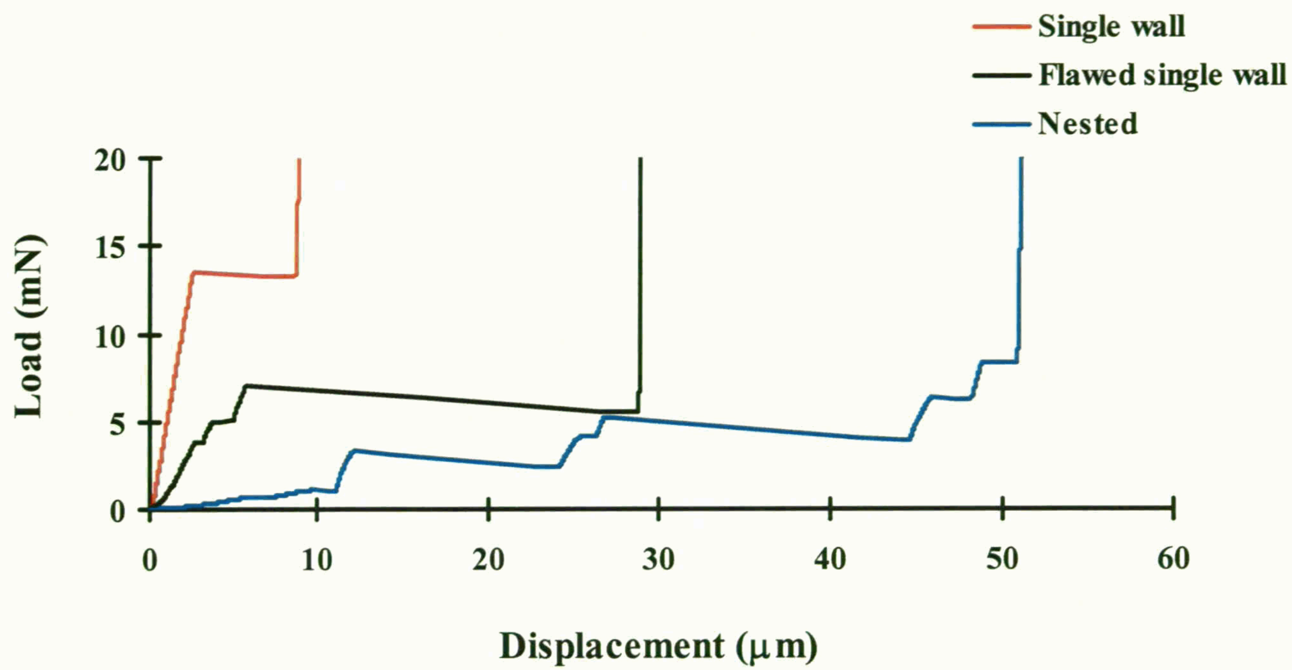


Parameters from nanocompression

- From graph:
  - Vertical diameter, ϕ_v
  - Failure properties
    - Displacement, δ_f
    - Load, P_f
  - Pseudo-stiffness, k
- Define:
  - Compressive strain
    - $\varepsilon_c = \delta_f / \phi_v$
  - Fracture energy
    - $E_f = 0.5\, P_f \delta_f$



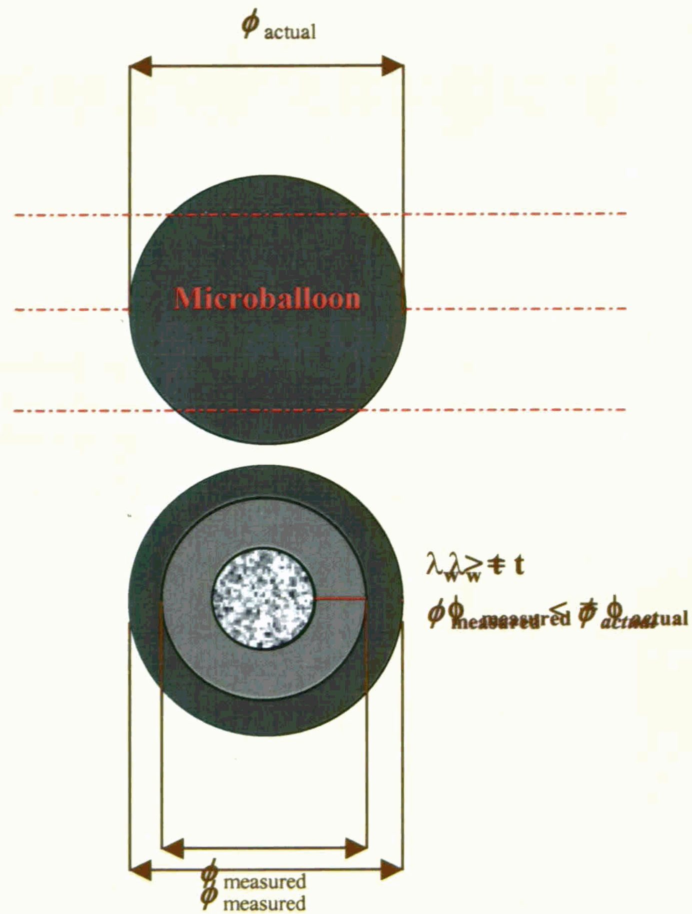
Typical nanocompression curves



- Categorize MBs according to curve shape
  - Single wall (SW)
  - Flawed single wall (FSW)
  - Nested (N)

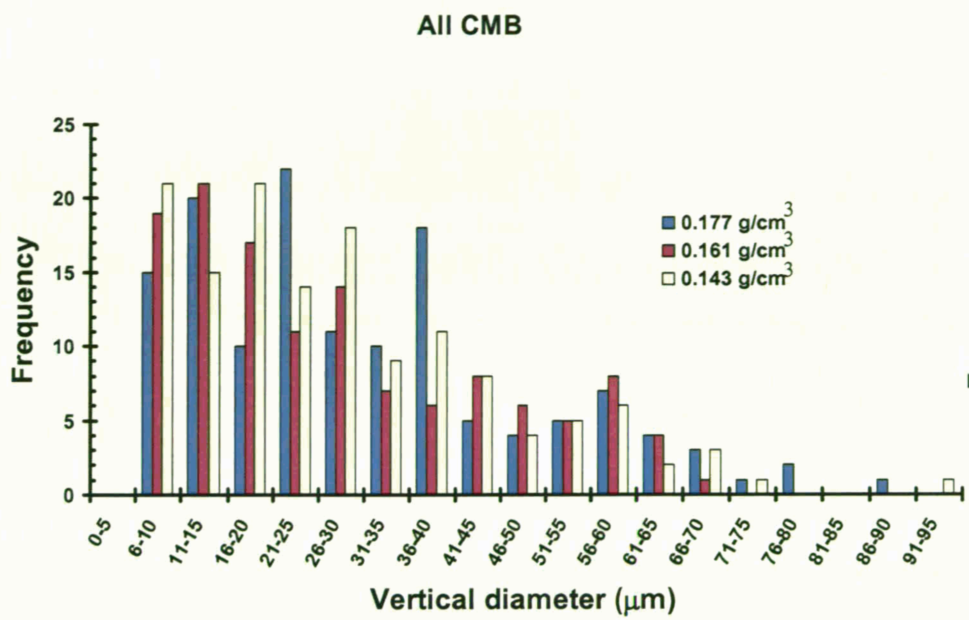
Procedure: Wall thickness measurement

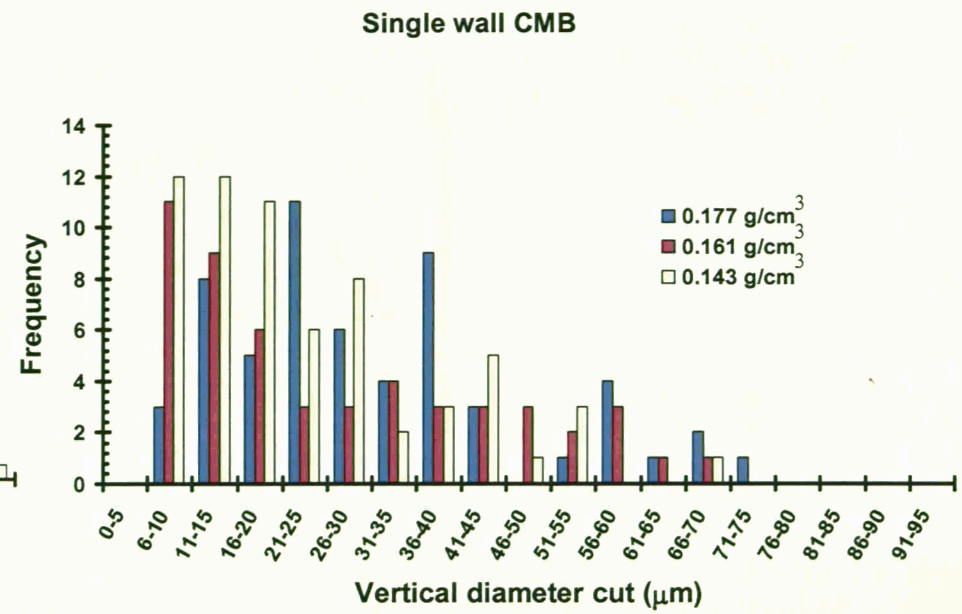
- Mount individual CMB in epoxy
- Polish
- Take random images of CMB cross-sections
- Statistically correct cross-sectional measurements
  - $\phi_{actual} = 1.5\,\phi_{measured}$
  - $\bar{\lambda}_{wall} = \dfrac{4t\left(t^2 - 1.5t\phi + 0.75\phi^2\right)}{3\left(t^2 - t\phi + 0.5\phi^2\right)}$
    - ϕ = diameter
    - t = actual thickness



Results & Discussion

Diameter distributions of CMB tested in compression





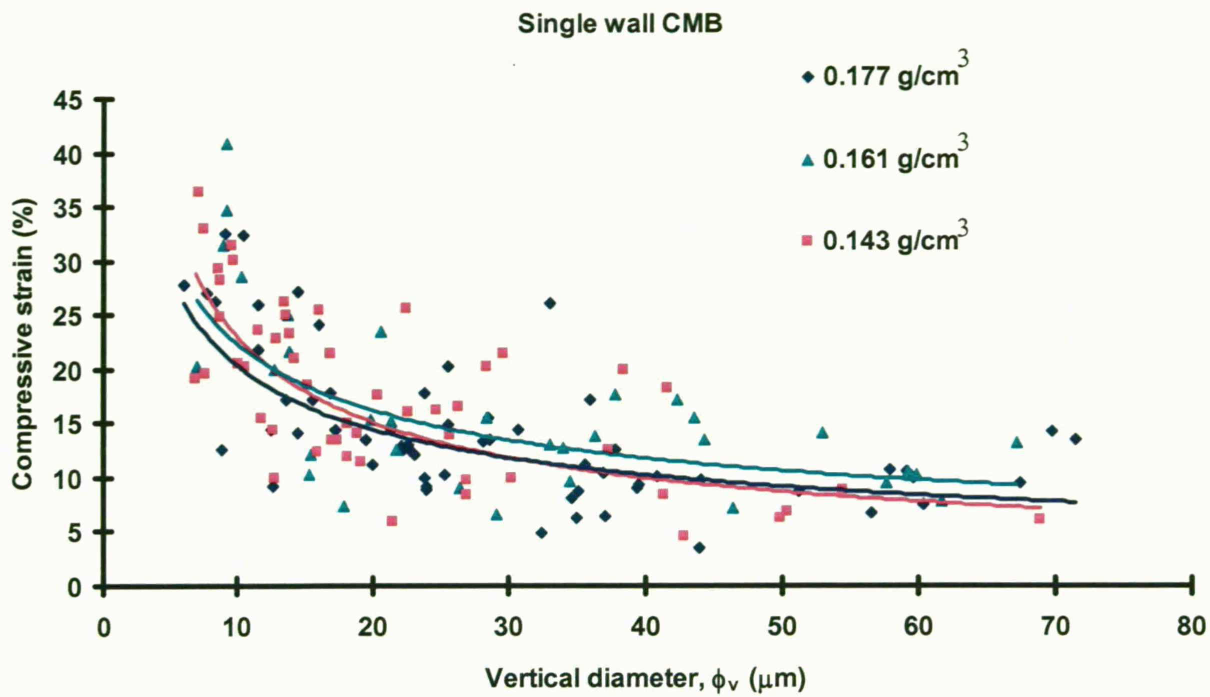
- Polymeric MB precursors usually have log normal distribution
  - CMB should be log normal also
  - The distribution tested may be typical, despite non-random sampling

Average failure properties

Single Wall Microballoons: Failure properties				
Tap density	**Failure Load, P_f (mN)**	**Compressive strain, ε_c (%)**	**Fracture energy, E_f (nJ)**	**Pseudo-stiffness, k (kN/m)**
0.143g/cm^3	11.44	17.66	22.22	3.54
0.161 g/cm^3	12.90	18.69	29.58	3.55
0.177 g/cm^3	14.65	16.48	27.49	4.73
Nested Microballoons: Ultimate failure properties				
Tap density	**Failure Load, P_f (mN)**	**Compressive strain, ε_c (%)**	**Fracture energy, E_f (nJ)**	**Pseudo-stiffness, k (kN/m)**
0.143g/cm^3	7.99	52.91	23.45	1.88
0.161 g/cm^3	10.72	57.81	19.89	3.06
0.177 g/cm^3	12.75	40.19	24.14	3.54
Nested Microballoons: Initial failure properties				
Tap density	**Failure Load, P_f (mN)**	**Compressive strain, ε_c (%)**	**Fracture energy, E_f (nJ)**	**Pseudo-stiffness, k (kN/m)**
0.143g/cm^3	2.71	12.33	6.25	0.77
0.161 g/cm^3	3.08	11.36	5.36	1.16
0.177 g/cm^3	3.89	10.80	8.20	1.10

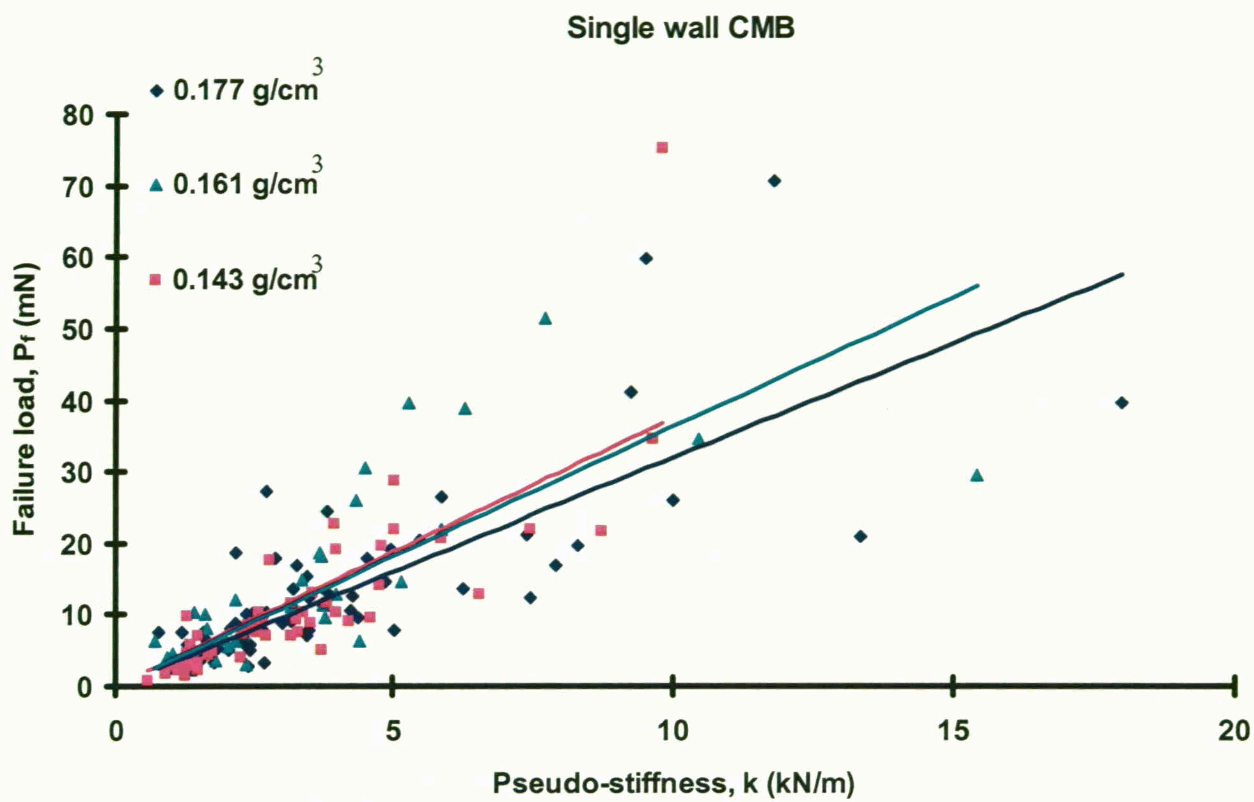
- CMB from higher tap densities have higher average failure properties
  - Applies for nested and single wall MB
- Nested microballoons fail at consistently lower values than single wall MB
  - Inferior properties to single wall

Compressive strain vs. diameter



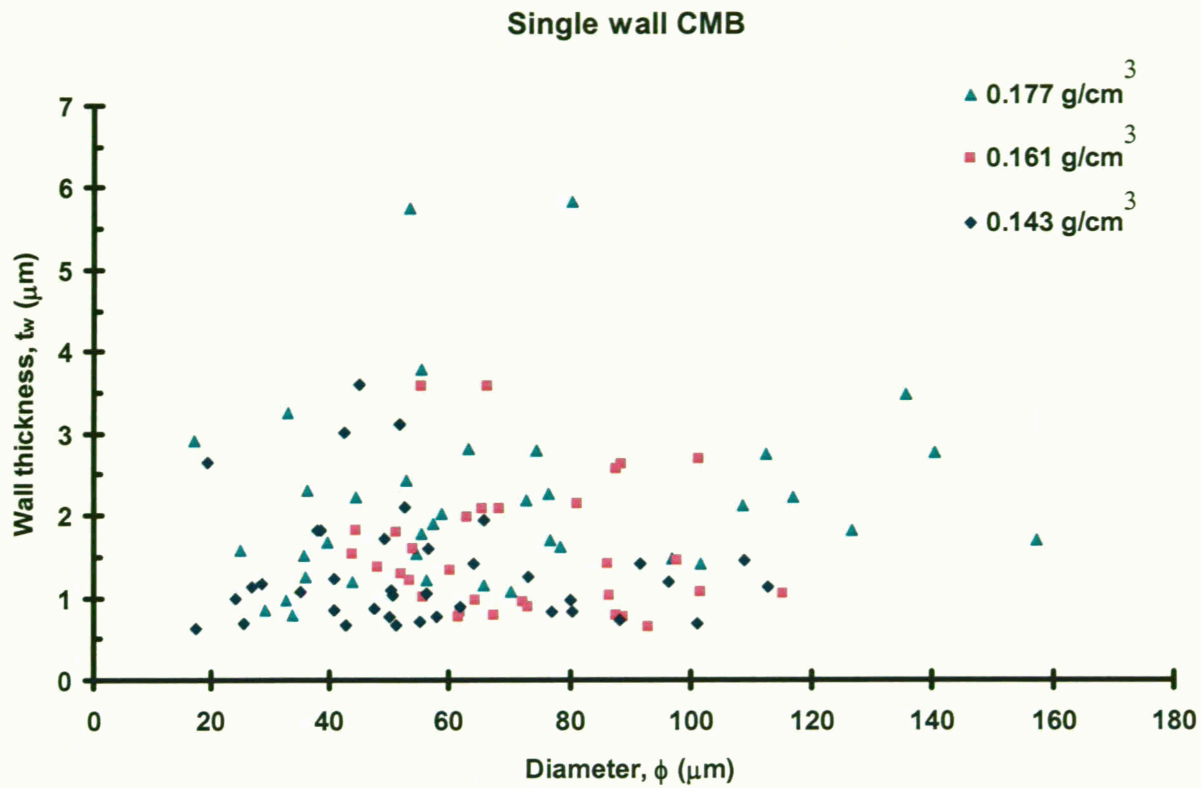
- Compressive strain proportional to inverse square root of CMB diameter
- Essentially independent of tap density

Failure load vs. pseudo-stiffness



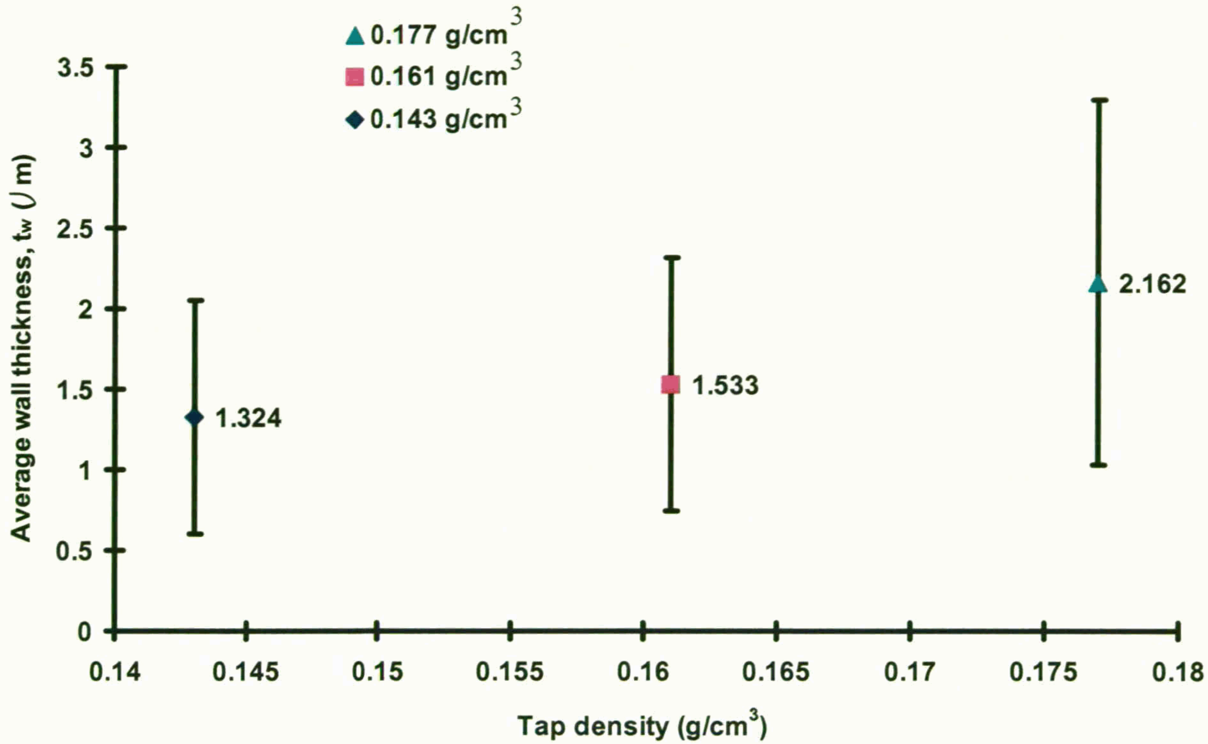
- Increased failure load = increased pseudo-stiffness
- Weak trend toward increasing k with higher tap density CMBs
  - $k = f(E, t, \phi)$

Wall thickness: Trends with CMB diameter



- No apparent trend with diameter for any tap density
- Best approach may be to use average wall thickness

Wall thickness: Average values



- General trend → increasing wall thickness with increasing tap density
  - Note: high degree of overlap in the populations
  - Not statistically different populations
- Most CMB wall thicknesses are within 1 standard deviation of the average

Summary

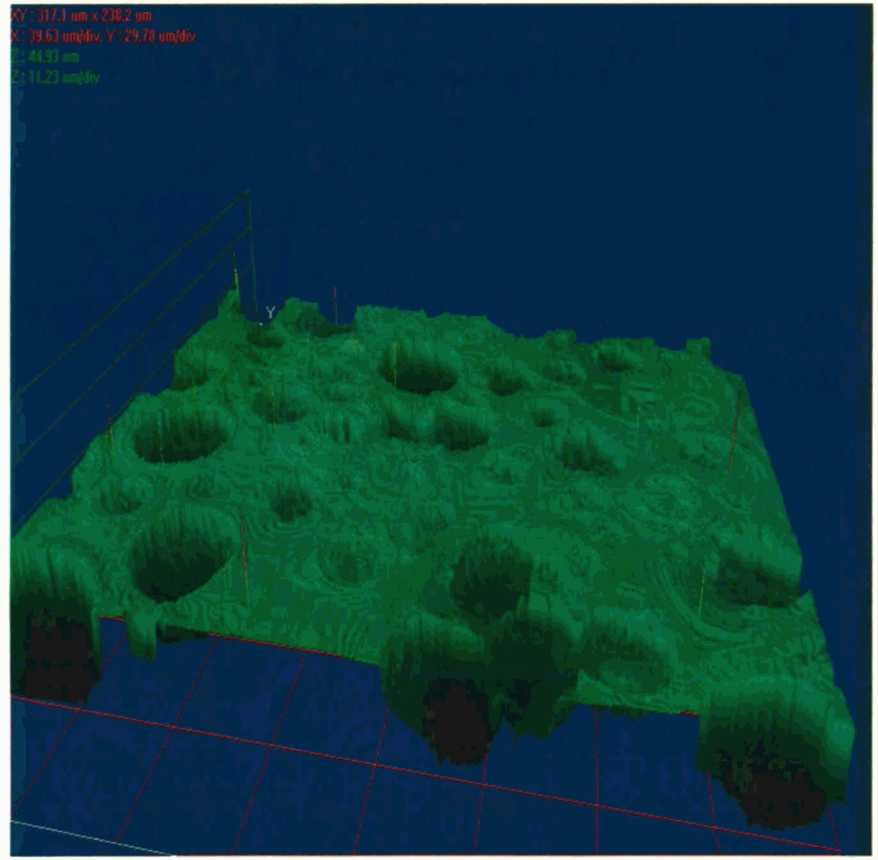
- As tap density increases:
  - Average failure load increased
  - Average wall thickness increased
  - Average pseudo-stiffness increased
- Average failure loads, fracture energies, and pseudo-stiffnesses for nested CMB are less than those for single wall CMB
  - Especially true for initial compartment failure
- Failure load vs. pseudo-stiffness:
  - Increasing failure loads yield increasing pseudo-stiffness, for all tap densities
  - At a constant failure load:
    - Pseudo-stiffness increases as tap density increases
- Compressive strain vs. diameter
  - Strain proportional to inverse square root of diameter
  - No trend with tap density

Conclusions

- What does this mean?
  - Increase in thickness with tap density could account for
    - Increased stiffness at a constant failure load
    - Increased average load and stiffness
    - Overlap in standard deviation of thickness measurements
      - Accounts for overlap in other properties
  - Single wall CMB exhibit superior properties to nested CMB
    - Greater wall thickness variation
    - More flaws
  - k definitely f (E, ϕ, t)
    - Could we define this with more data?

Future work

- Other tap densities
- Coated microballoons
  - APO-BMI coating to simulate behavior in foam
- Fragment capture
  - Wall thickness of actual CMB tested
- Video Nanocompression test
- FE simulation
- Interferometry
  - Direct measure of wall thickness & diameter



Acknowledgements

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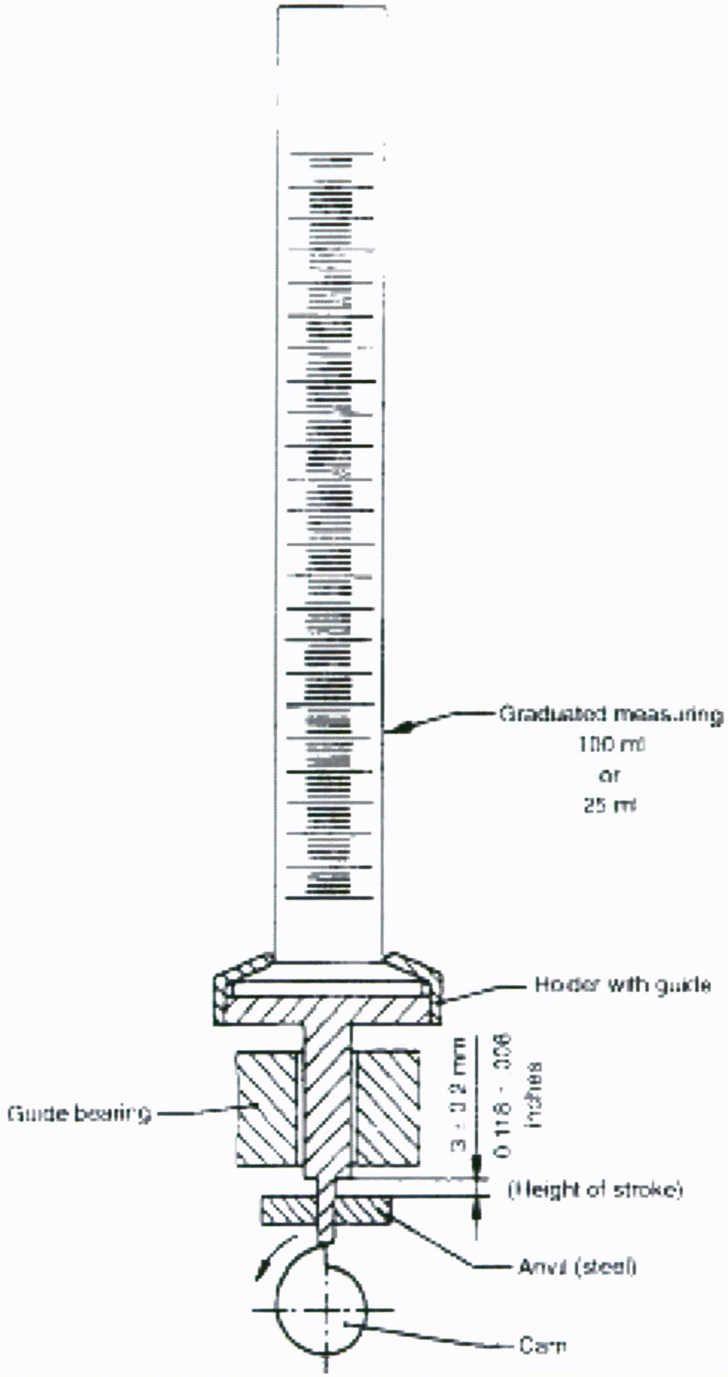

M B 527 - 93

- Standard for the measurement of tap density of metallic powders and compounds
- Correlates to bulk foam properties
- Method
  - Place 50 ± 0.2 g CMB into 100 ml graduated cylinder
  - Subject to 3000 taps to settle powder
  - Pt = M/V